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U1S S1814 S2271 S2293

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WPI Accession no 95-094233 & JP070018442 A

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INT CL^6 C23C 14/24 14/28 14/30 14/56
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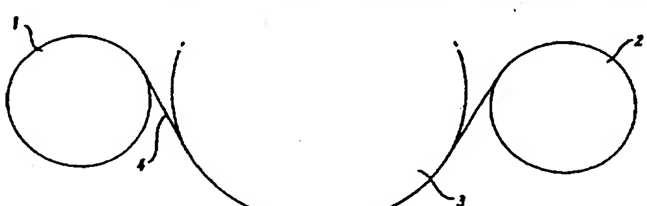
(54) Abstract Title

Vacuum deposition on a moving web using a radiant heat supply

(57) In apparatus for a method of coating a web, a deposition station is disposed in a vacuum chamber with means for supplying the web to unwind roll 11, and recovering the web from, rewind roll 2, the station. The station comprises a boiler 5 in which material to be sublimed or evaporated is deposited and means for subjecting this material to radiant heat by means of a resistance heated boat 7 housed in compartment 6 to produce sublimation or evaporation and deposition of the sublimed material onto the web. Compartment 6 is contained within the main body 8, which contains the material to be sublimed or evaporated, of the boiler 5. The sublimed material may be zinc sulphide and the coating produced on the web may be optically monitored by means of an optical spectrometer.

The web may be initially coated with aluminium or copper and materials deposited may be zinc sulphide, silica, copper or silver. The reflectivity of the coating may be monitored and the coating thickness may be controlled. The boiler may comprise a main body formed of TiB_2, W, Ta or Mo.



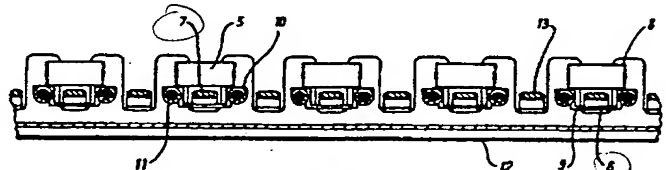


FIG. 2

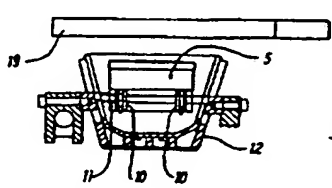


FIG. 1

At least one drawing originally filed was informal and the print reproduced here is taken from a later filed formal copy.

The claims were filed later than the filing date but within the period prescribed by Rule 25(1) of the Patents Rules 1995.

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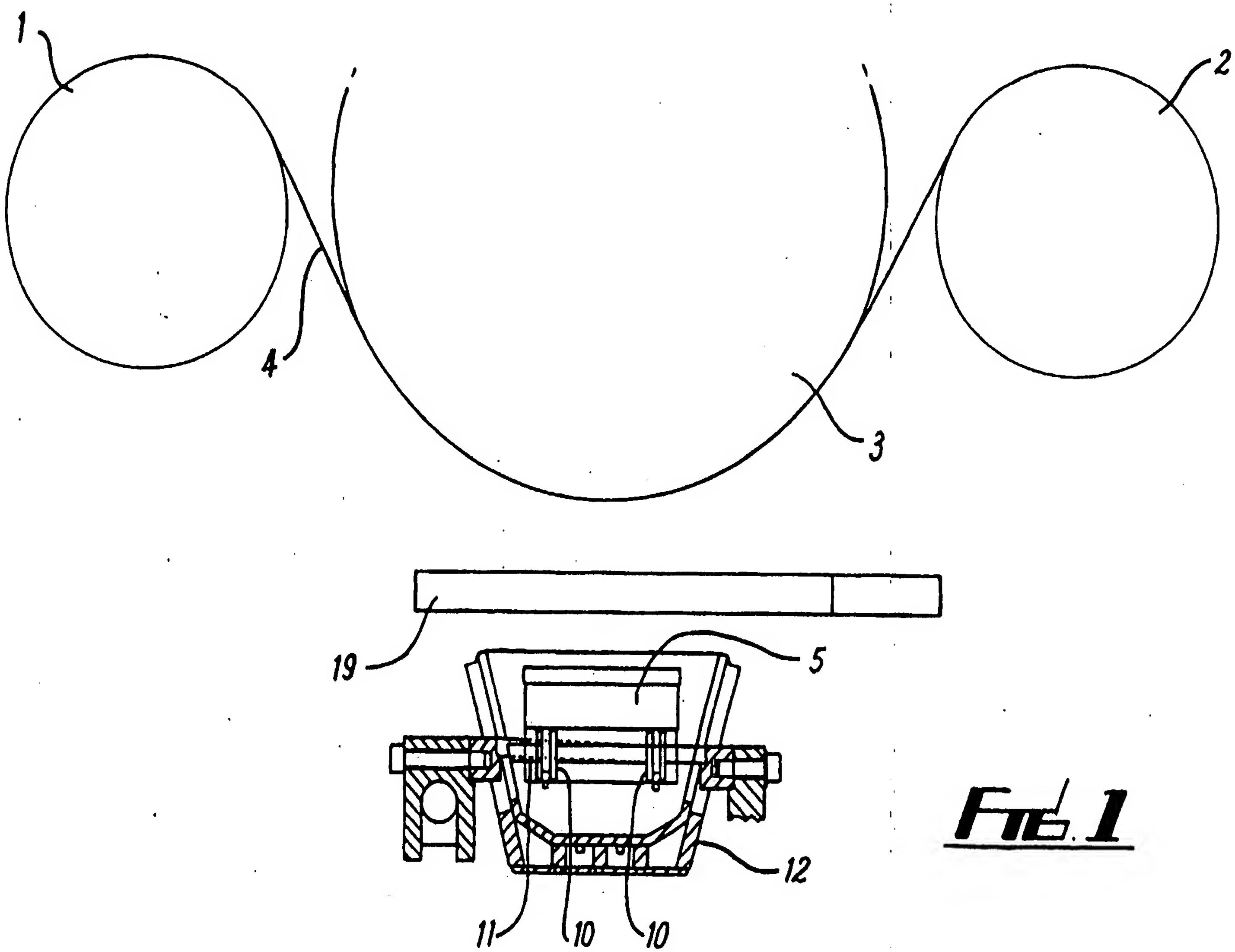

FIG. 1

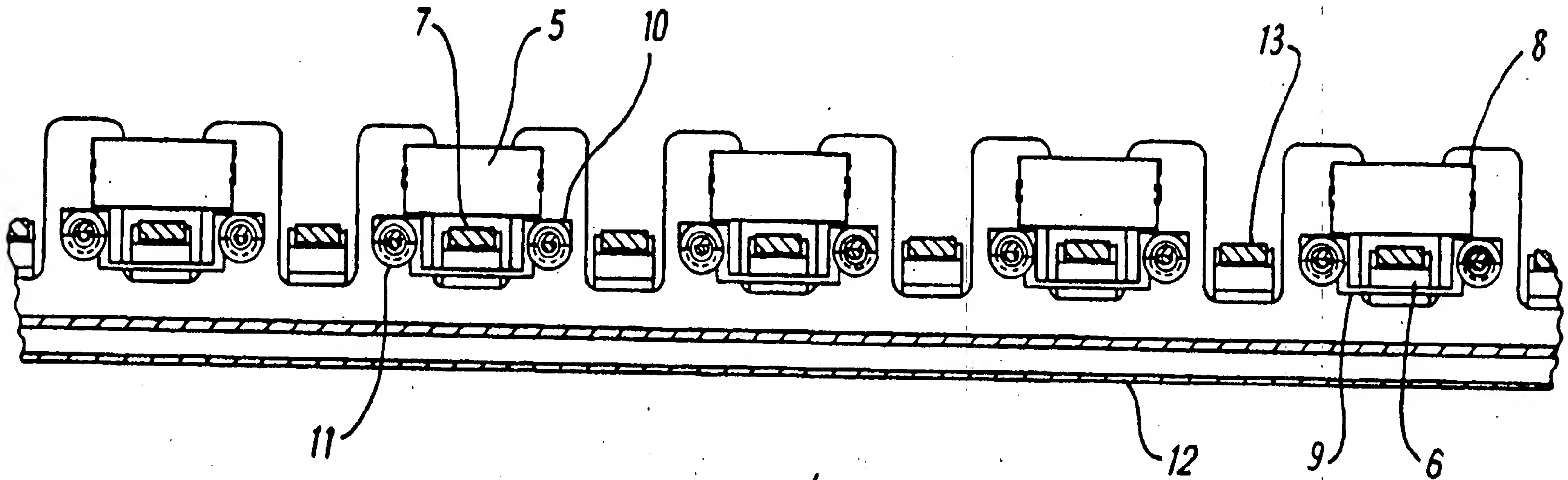

Fig. 2

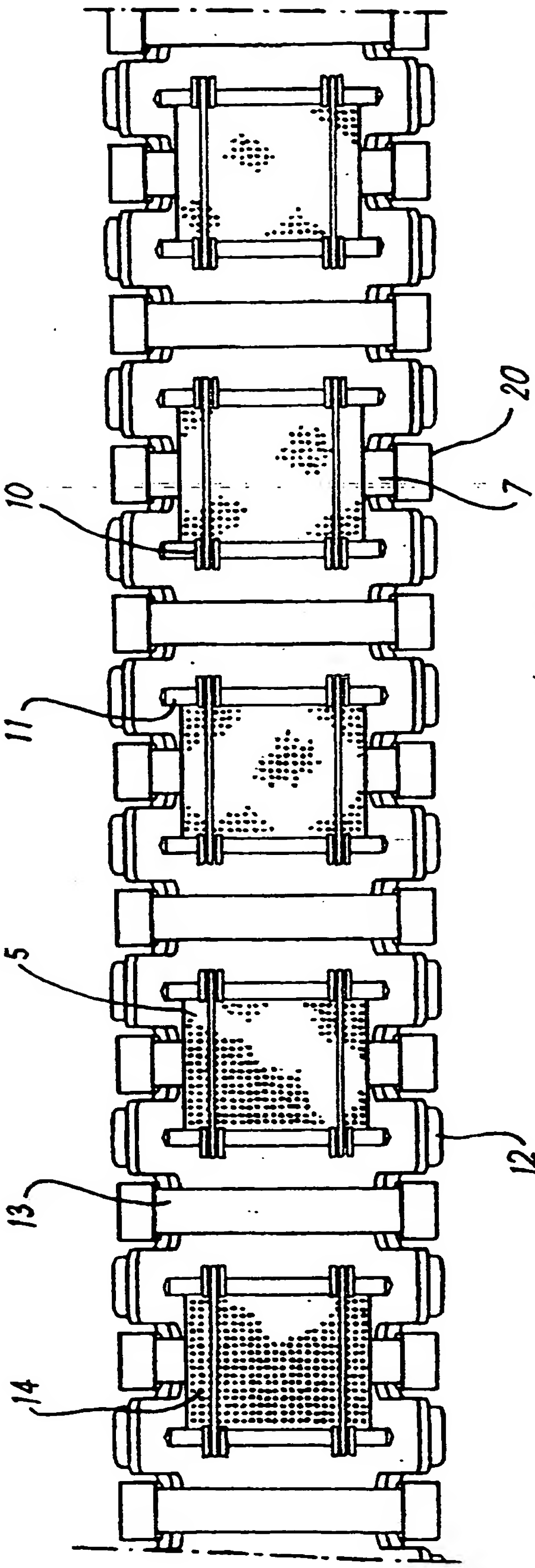

FIG. 3

A VACUUM PROCESS FOR DEPOSITING ZINC SULPHIDE AND OTHER COATINGS ON FLEXIBLE MOVING WEB

The present invention relates to a vacuum process and apparatus for depositing zinc sulphide and other coatings on flexible moving web.

Over the past few years holograms and diffraction gratings have grown into a complex business for security cards, product packaging and decorative wrapping. Further development was carried out to prevent counterfeiting of holographic security cards, banknote and security labels. The rapid advances in holographic technology required the utilization of advanced optical coatings and further development of these methods to reduce production costs. Consequently, there has been an increasing interest in zinc sulphide (ZnS) for its high refractive index and wide wavelength passbands. When applied to films such as polyester it produces a semi-transparent coating that is reflective when viewed at selected orientation. This material is inexpensive as compared to others such as titanium dioxide and can be encapsulated and used in I.D. cards, credit cards and product labels as well as packaging. At present there are various methods for evaporating zinc sulphide including direct resistance heating, induction heating and electron beam evaporation. However, there are problems and disadvantages associated with each process. For example, direct resistance heating requires expensive boats that use excessive high

power and energy consumption. In this case evaporation boats have to be replaced regularly due to breakdown and are not designed to receive a sufficiently large charge of ZnS for coating a roll of plastic film. The other problem is the interaction of ZnS with boat electrical resistance which affects the stability of the applied power and hence, the evaporation rate. If tantalum boats are used, the hear required to sublime the ZnS for an extended period eventually oxidises and breaks the tantalum. Heavier gauge boats require higher power and the shape of the boats would result in a discontinuity in electrical resistance and heat build-up leading to the breakage of the contacts. On the other hand, induction heating (see US patent 5695808) requires expensive equipment and a major modification to the conventional vacuum coater to convert it for ZnS deposition. Induction heating requires high energy, large power supply for the generation of RF and induction coils around each crucible. This makes the process more expensive. The utilization of electron beam evaporation would result in an irregular sublimation of ZnS material and the formation of a large hole in the area where the beam hits the material. Scanning the electron beam would result in materials jumping out of the crucible. The sublimation rate in this case is unsteady and forms thick and thin patches of ZnS coating.

According to the one aspect of the invention there is provided apparatus for depositing a coating on a flexible moving web comprising a

housing defining a vacuum chamber, means for creating a vacuum in the chamber, a deposition station disposed in the chamber, means for supplying web to the deposition station and means for receiving web from the deposition station, the deposition chamber comprising a boiler in which in operation material to be sublimed may be deposited and means for subjecting material to be sublimed to radiant heat to produce sublimation of material in the boiler.

According to another aspect of the present invention there is provided a method of depositing a coating on a web including the steps of supplying the web to a deposition station under vacuum conditions, supplying material to be sublimed to the deposition station and subjecting the material to radiant heat to produce sublimation of the material and deposition of the sublimed material on the web.

In a preferred embodiment of the invention the boiler is electrically and thermally isolated. The evaporation (sublimation) boiler is indirectly heated from a conventional boat which is located underneath the main body. During operation the boiler is filled with ZnS or other material tablets or pellets then covered with a top perforated baffle to prevent material jumping out of the boiler during sublimation. The boat/boiler assembly is clamped between two water cooled copper contacts inside a standard vacuum coater which contain a supply roll of the polymer film and a take-up

roll. After evacuating the chamber, the boats are heated up by conventional resistance heating. Eventually, the sublimation boiler is heated up by radiant heating from the boat till it reaches the required temperature to start sublimation of ZnS. The ZnS vapour is then deposited on the polymeric film passing in proximity of the box to form a layer. The coating reflectivity is monitored visually or by using an optical spectrometer and the coating thickness is controlled by adjusting source power and film speed.

In order that the invention may be more clearly understood an embodiment thereof will now be described, by way of example with reference to the accompanying drawings in which:-

Figure 1 shows diagrammatically a partial view in cross-section of a vacuum coating apparatus,

Figure 2 is a side view of the part of the apparatus of figure 1 to a smaller scale,

Figure 3 is a top view of the part of the apparatus shown in figure 2.

Referring to figure 1, a zinc sulphide (ZnS) coating is deposited on a moving polymer or other web 4 inside a vacuum chamber. The polymer web may be initially coated with a sufficient thickness of aluminium, copper or other metals. For this purpose a two zone conventional vacuum web coater is used. The coater has a winding system composed of independently driven unwind roll 1, rewind roll 2 and cooled process drum

3 to move the polymer or other web 4 at various speeds under controlled tension. An evaporation source consists of a specially designed evaporation boiler 5. A movable shutter 19 is interposed between the boiler 5 and process drum 3. This is moved to allow evaporant access to the web when the temperature of the boiler has reached its operational level.

Figure 2 shows the arrangement of the evaporation boiler 5 inside the vacuum coater. The evaporation boiler 5 is made of molybdenum sheet although other high refractory materials such as tantalum or tungsten graphite or other high melting point material can be used. The evaporation boiler 5 consists of two parts; a small compartment 6 to house a resistance heated boat 7 and a main body 8 to contain material to be sublimed or evaporated. Resistance heating is by high current lower voltage power. For Zinc Sulphide (ZnS) sublimation, the resistance heated boat 7 can be made of intermetallic materials such as titanium diboride or ceramics. For other high melting evaporant, the boats can be made of refractory materials such as tungsten, tantalum or molybdenum. The boat is electrically isolated from the boiler 5 and clamped in position by means of a water cooled clamp 20. The boiler 5 rests on two brackets 9 made of molybdenum, ceramics or other refractory materials. The bracket 9 sit on high temperature ceramic insulators 10 which are inserted on top of steel bars 11. The bars 11 rest on the sides of water cooled copper trough 12. The lower compartment of

the boiler 6 captures radiant heat from the electrically heated boat 7 and increases the boiler temperature to 1200°C or higher.

In the present embodiment five boilers placed at a distance of 200mm from each other are used as shown in figure 3 to coat a 640mm wide film. More boilers can be used for wider films. Extra boats 13 can be used for higher temperatures. The boilers are covered with perforated top baffles 14.

These baffle prevent the ZnS material from jumping out of the boilers. They can be removed for faster deposition. This arrangement produces a uniformity of ±5% across the width of the polymeric film at a film speed of 35-40m/min. The uniformity can be further improved by adjusting the spacings between boilers. Coating reflectance achieved with this setup is 40% at an optical wavelength of 500-550nm. Boiler size used is 150mm long x 60mm wide x 40mm deep but larger sizes can also be used. Each boiler is filled with 500g to coat a total film length of 2000m or more.

The vacuum chamber can be provided with two light sources with a width wider than the film width to facilitate viewing the web through the chamber view port. One light source can be used behind the moving film before the rewind zone to view the transmitted light through the coated film. The other light source can be fixed on the chamber wall above the

view port to provide a reflected light. For a thickness of 500-600Å, the film appears highly reflective with a light yellow tint. Above this thickness, the film exhibits variable colours from gold yellow to red depending on the thickness of Zinc Sulphide (ZnS) coating. The particular colours are visible when the film speed slows down or the evaporation rate increases by increasing boat power.

By adjusting boilers spacing, boats power and film speed the uniformity of the coating across the film width can be enhanced to better than ±5%. The present invention provides means to control coating reflectivity by using Ocean Optics SQ2000 optical spectrometer in the visible region. The reflectivity over a waveband of 300nm to 800nm can be displayed on a computer screen. Alternative optical or resistivity probes can also be used for this purpose. The probes can be fixed on a long bar facing the film as it leaves the coating zone and before rewinding. The distance between the probe and film can be adjusted and can be made within a few millimetres. Before evacuating the chamber each probe is calibrated using a standard mirror to give 100% reflectivity. For a further control, thermocouples can be attached to each boiler to control the sublimation temperature. This temperature is maintained throughout the coating of the web by adjusting the power of individual boats. After achieving the required reflectance, film speed and boiler's temperature are

then maintained throughout the coating. As the subliming material starts to run out the film/web speed can then be reduced to get back the same reflectance. The present invention is not restricted to Zinc Sulphide ZnS. Other coating materials including silicon oxide, copper, silver, indium and indium tin oxide can also be evaporated using this invention. Plasma assisted deposition can also be carried with this setup.

The above described arrangement may be retrofitted to an existing conventional vacuum coater. The coating produced can be monitored either by visually observing surface reflectance or by using an optical monitor. Coating thickness and reflectance may be controlled by adjusting the electrical power to heat the boats or the speed of the web. Coating quality can be improved by running the process drum at room temperature. A plasma discharge source can be fitted between the evaporation boiler and process drum to improve coating characteristics. Other materials than zinc sulphide such as silicon dioxide, copper, silver can be evaporated using the above arrangement. The coating may be applied to clear or embossed polymeric substances as well as to paper or metal substrates. A feeding mechanism such as vibrating pellet feeder can be used to feed the boilers for a large roll of web. The feeder can be fixed to a moving plate and can be moved forward to a location next to the boiler to drop zinc sulphide pellets or tablets. The feeder can be shielded from the vapour stream during

the coating process.

It will be appreciated that the above embodiment has been described by way of example only and that many variations are possible without departing from the scope of the invention. For example, although the boilers are shown as generally rectangular, they may be circular.

CLAIMS

1. Apparatus for depositing a coating on a flexible moving web comprising a housing defining a vacuum chamber, means for creating a vacuum in the chamber, a deposition station disposed in the chamber, means for supplying web to the deposition station and means for receiving web from the deposition station, the deposition chamber comprising a boiler in which in operation material to be sublimed or evaporated may be deposited and means for subjecting material to be sublimed or evaporated to radiant heat to produce sublimation or evaporation of material in the boiler.

2. Apparatus as claimed in claim 1, in which the boiler is electrically isolated.

3. Apparatus as claimed in claim 1 or 2, in which the boiler is thermally isolated.

4. Apparatus as claimed in claim 1, 2 or 3, in which the boiler is indirectly heated.

5. Apparatus as claimed in claim 1, 2, 3 or 4 in which the boiler comprises a main body adapted to contain material to be sublimed or evaporated above a boat which is heated in operation.

6. Apparatus as claimed in claim 5, in which resistance heating is provided to heat the boat.

7. Apparatus as claimed in claim 6, in which the resistance heated boat is made of intermetallic material.

8. Apparatus as claimed in claim 7, in which the intermetallic material is titanium dioboride or ceramics.

9. Apparatus as claimed in claim 5 or 6, in which the boat is made of a refractory material.

10. Apparatus as claimed in claim 9, in which the refractory material is tungsten, tantalum or molybdenum.

11. Apparatus as claimed in any preceding claim, in which a baffle is provided on the boiler operative to prevent material to be sublimed or evaporated jumping out of the boat.

12. Apparatus as claimed in claim 11, in which the baffle is perforated.

13. Apparatus as claimed in claim 11 or 12, in which the baffle is moveable.

14. Apparatus as claimed in any preceding claim, in which a moveable shutter is disposed between the boiler and web operative to be moved to allow sublimed or evaporated material access to the web when the boiler has reached its operational level.

15. Apparatus as claimed in any preceding claim, in which a plurality of boilers is disposed across the width of a web to be coated.

16. Apparatus as claimed in any preceding claim, in which means are

provided for monitoring the coated web.

17. Apparatus as claimed in claim 16, in which the means for monitoring comprises an optical spectrometer.

18. A method of depositing a coating on a web including the steps of supplying the web to a deposition station under vacuum conditions, supplying material to be sublimed or evaporated to the deposition station and subjecting the material to radiant heat to produce sublimation or evaporation of the material and deposition of the sublimed or evaporated material on the web.

19 A method as claimed in claim 18, in which a zinc sulphide coating is deposited on the web.

20. A method as claimed in claim 18 or 19, in which the web is initially coated with aluminium, copper or other metal.

21. A method as claimed in claim 18, 19 or 20, in which the reflectivity of the coating on the web is monitored.

22. A method as claimed in claim 21, in which the coating thickness is controlled in dependence upon the reflectivity monitored.

23. A method as claimed in claim 22, in which the coating thickness is controlled by controlling the supply of radiant heat to the boiler.

24. Apparatus for depositing substantially as hereinbefore described with reference to the accompanying drawings.

25. A method for depositing substantially as hereinbefore described with reference to the accompanying drawings.

Application No: GB 9816211.8
Claims searched: 1-25

Examiner: Pete Beddoe
Date of search: 12 August 1999

Patents Act 1977
Search Report under Section 17

Databases searched:

UK Patent Office collections, including GB, EP, WO & US patent specifications, in:

UK Cl (Ed.Q): C7F (FCAV, FCAX, FCVE, FCVL, FCVM, FCVX, FCXE, FCXL, FCXM, FCXX, FEAA, FEH)

Int Cl (Ed.6): C23C (14/24, 14/28, 14/30, 14/56)

Other: Online; WPI, EPODOC, JAPIO

Documents considered to be relevant:

Category	Identity of document and relevant passage		Relevant to claims
X	GB 2306512 A	(BALZERS) see esp page 4 lines 20-25 & fig	1,18 at least
X	EP 0794266 A1	(ALUSUISSE) see esp claim 1, col9 line 39 - col10 line 47, figs & English abstract	1,18 at least
X	EP 0784102 A1	(ALUSUISSE) see esp claim 1, example, figs & English abstract	1,18 at least
X	DD 160748 A	(STEINFELDER) see esp claim 1, example at p4 & WPI English abstract	1,18 at least
X	DE 4442733 A	(ARDENNE) see esp col1 line 54 - col2 line 59, claim 1, figs & WPI English abstract	1,18 at least
X	US 5679410	(MATSUSHITA) see esp col3 line 41 - col4 line 44 & figs	1,18 at least
X	US 5616362	(ANDRITZ) see esp col4 lines 3-25 & figs	1,18 at least
X	US 5614273	(FRAUNHOFER) see esp col6 line 48 - col7 line 52 & figs	1,18 at least

X Document indicating lack of novelty or inventive step
Y Document indicating lack of inventive step if combined with one or more other documents of same category.
& Member of the same patent family

A Document indicating technological background and/or state of the art.
P Document published on or after the declared priority date but before the filing date of this invention.
E Patent document published on or after, but with priority date earlier than, the filing date of this application.

Application No: GB 9816211.8
Claims searched: 1-25

Examiner: Pete Beddoe
Date of search: 12 August 1999

Category	Identity of document and relevant passage	Relevant to claims
X	US 5302208 (LEYBOLD) see esp col2 lines 39-52 & figs	1,18 at least
X	US 5239611 (WEINERT) see esp col3 lines 9-33, claim 1 & fig 2	1,18 at least
X	US 4959524 (RUDNAY) see esp col5 lines 5-54 & claim 1	1,18 at least
X	WPI Accession no 95-094233 & JP 7018442 A (MATSUSHITA) see abstract	

X	Document indicating lack of novelty or inventive step	A	Document indicating technological background and/or state of the art.
Y	Document indicating lack of inventive step if combined with one or more other documents of same category.	P	Document published on or after the declared priority date but before the filing date of this invention.
&	Member of the same patent family	E	Patent document published on or after, but with priority date earlier than, the filing date of this application.